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ABSTRACT

High temperature stability of the microstructure of Nb-1Zr sheet containing 0.1 and 0.06 wt.%C was studied as affected by processing and prolonged 1350-K exposure with and without applied stress. Sheets were fabricated by cold rolling bars that were single-, double- or triple-extruded at 1900 K. Creep samples were double-annealed (1 h @ 1755 K + 2 h @ 1475 K) prior to testing at 1350 K for 10,000 - 34,500 h. The microstructures of the as-cast, extruded, rolled, DA and crept samples were characterized using various metallographic and analytical methods. The precipitates were rather coarse Nb₂C initially, but transformed to finer ($\leq 1 \mu\text{m}$) carbides of (Zr,Nb)C with each subsequent high temperature process. The grain size, and the relative amount and morphology of (Zr,Nb)C were found to be affected by the number of extrusions and to some extent by C-content. However, the microstructures of all the crept samples were similar with (Zr,Nb)C distributed throughout the matrix indicating that prolonged exposure to 1350 K gave rise to complete transformation of Nb₂C to (Zr,Nb)C regardless of the processing history. These and other observations are presented with the emphasis on the correlation between processing, microstructure and creep properties.

INTRODUCTION AND BACKGROUND

Advanced power systems with a nuclear reactor as the primary heat source are being developed to provide electricity for future space missions. One of the critical concerns is the selection of the proper materials to meet the rather stringent design requirements of the reactor system. These include a full-power (100 kW) operating life of 7 years at 1350-1450 K and 5-25 MPa in a liquid alkali metal environment with total creep strain of less than 2%^{1,2}. Nb-Zr alloys with and without C were selected over other refractory metal alloys primarily because of their resistance to liquid alkali metal corrosion, ease of fabrication and relatively low densities. In particular, Nb-1Zr (all compositions in wt.%) was chosen for ground demonstration of the power system³ and Nb-1Zr-0.1C, an alloy developed in 1960s⁴, was suggested to provide additional design margins⁵. Reactor test loops with components made from both alloys have been tested successfully in liquid lithium for up to 3200 hours⁶. However, recent work⁷⁻¹⁰ showed that the creep resistance of the precipitate-hardened Nb-1Zr-C alloys is superior to that of the solid solution-strengthened Nb-1Zr. Based upon weight limits, it was also clear that a Nb-1Zr-C alloy must be used in the parts of the power system where the stresses are expected to approach or exceed 10 MPa at 1350 K when a service life of 7 years or more is required¹⁰.

The strength and creep resistance of Nb-Zr-C alloys is due to the carbides of Nb and/or Zr; therefore, it is important to have a complete understanding of their precipitation sequence, morphology and high temperature stability. A number of studies have been reported on microstructural characterization of the Nb-Zr-C alloys¹¹⁻²³, but only a few investigated the effects of processing and long-term exposure to elevated temperatures^{10,22,23}. This paper deals with the characterization and long-term stability of the microstructure of Nb-1Zr sheets containing 0.06 and 0.1C as affected by processing and carbon content with the emphasis on precipitate composition and morphology.

EXPERIMENTAL

Samples were taken from the materials before and after each process, and the condition of each as examined in this study is listed in Table I. Uniaxial creep tests were conducted on 1-mm thick sheets fabricated from vacuum arc-melted (VAM) ingots by a combination of hot extrusion and cold rolling operations as indicated. Two Nb-1Zr-0.06C samples were creep tested, one in double-annealed (DA) and the other in double-annealed and aged (DA/AGE) condition. A sample from each of the three Nb-1Zr-0.1C sheets was also creep tested after the double-anneal heat treatment. All the creep tests were carried out in vacuum (10^{-6} Pa or better pressure) at 1350 K for times ranging from about 10,000 h to 34,500 h. The ends (E) of each of the crept samples were assumed to be heat treated without applied stress, because the regions beyond the grips of the uniaxial creep samples underwent no measurable deformation. This allowed the assessment of the effect of stress on the microstructure during high temperature exposure.

The as-received samples were chemically analyzed to verify initial composition. Additionally, all the heat-treated and crept samples were analyzed to monitor the loss or pick up of interstitial impurities (O, N, C).

Table I. Processing history and condition of each sample from sheets of Nb-1Zr-0.06C (LC-samples) and Nb-1Zr-0.1C (064-samples).

SAMPLE	NUMBER OF EXTRUSIONS ^(a)	HEAT TREATMENT/CONDITION
LC	1	AS COLD ROLLED
LC-DA	1	DA ^(d)
LC-DA10(E) ^(b)	1	DA + 34,500 h @ 1350 K
LC-DA10(M) ^(c)	1	DA + 34,500 h @ 1350 K and 10 MPa
LC-DA/AGE	1	DA + AGE ^(e)
LC-DA/AGE10(E)	1	DA + AGE + 32,500 h @ 1350 K
LC-DA/AGE10(M)	1	DA + AGE + 32,500 h @ 1350 K and 10 MPa
064-INGOT		VACUUM ARC-MELTED INGOT
064A	1	AS COLD ROLLED (96%)
064A-DA	1	DA
064A-DA34(E)	1	DA + 18780 h @ 1350 K
064A-DA34(M)	1	DA + 18780 h @ 1350 K and 34.5 MPa
064B	2	AS COLD ROLLED (88%)
064B-DA	2	DA
064B-DA34(E)	2	DA + 15460 h @ 1350 K
064B-DA34(M)	2	DA + 15460 h @ 1350 K and 34.5 MPa
064C	3	AS COLD ROLLED (60%)
064C-DA	3	DA
064C-DA34(E)	3	DA + 9950 h @ 1350 K
064C-DA34(M)	3	DA + 9950 h @ 1350 K and 34.5 MPa

^(a) Number of hot (1900 K) extrusions prior to cold rolling of the sheet bars.

^(b) (E): Low-stress end portions of crept sample.

^(c) (M): Stressed gage section of crept sample. Total strain was <0.3% in each.

^(d) DA: Double-anneal, 1h @ 1755 K + 2h @ 1475 K, furnace-cooled after each step.

^(e) AGE: Heat treatment of 1000 h @ 1350 K followed by furnace cooling.

The sheet samples were examined in the as-polished condition by light microscopy and scanning electron microscopy (SEM) for precipitate morphology and distribution. The polished specimens were also etched by a solution of 30 ml lactic-15 ml nitric-5 ml hydrofluoric acids and then examined by light microscopy for grain size. A lineal intercept method²⁴ was used to determine the grain size. Phase extraction was performed on each specimen listed in Table I using a solution of 900 ml methanol-100 ml bromine-10 g tartaric acid with platinum as the catalyst. The residue obtained from each was analyzed by (1) x-ray spectroscopy for precipitate identification and (2) an inductively-coupled plasma (ICP) method for Nb and Zr contents. The double-annealed samples from the Nb-1Zr-0.1C sheets were also examined by transmission electron microscopy (TEM) to verify the results from X-ray analysis of the residue. Energy-dispersive X-ray spectroscopy (XEDS) using SEM was also performed on various residue samples to verify the trends observed from the ICP results.

RESULTS AND DISCUSSION

Chemical Analysis and Metallography

The chemical composition and grain size of the samples are given in Table II. These together with the microstructural observations are discussed below.

Table II. Chemical analysis and grain size for Nb-1Zr-0.06C (LC-) and Nb-1Zr-0.1C (064-) sheet samples.

SAMPLE	CHEMICAL COMPOSITION (wt.%)				GRAIN SIZE (μM)	ASPECT RATIO
	O ^(a)	N ^(a)	C ^(b)	Zr ^(c)		
LC	0.0078	0.0053	0.0630	0.90		
LC-DA	0.0034	0.0050	0.0500	0.90	25 \pm 4	1-3
LC-DA/AGE	0.0031	0.0030	0.0600	0.90	25 \pm 1	1-2
LC-DA10(E)	0.0080	0.0032	0.0640	0.90	21 \pm 3	1-2
LC-DA10(M)	0.0430	0.0026	0.0620	0.90	26 \pm 1	1-2
LC-DA/AGE10(E)	0.0120	0.0040	0.0640	0.90	27 \pm 2	1-2
LC-DA/AGE10(M)	0.0270	0.0035	0.0610	0.90	29 \pm 3	1-2
064-INGOT	0.0050	0.0025	0.0910	0.96		
064A	0.0035	0.0025	0.0900	0.93		
064A-DA	0.0028	0.0019	0.0921	0.95	110 \pm 14	1-2
064A-DA34(E)	0.0133	0.0026	0.0920	0.95	31 \pm 2	1-2
064A-DA34(M)	0.0220	0.0044	0.0980	0.95	32 \pm 4	1-2
064B-DA	0.0033	0.0022	0.0914	0.95	69 \pm 7	1-3
064B-DA34(E)	0.0176	0.0046	0.0890	0.95	39 \pm 4	1-2
064B-DA34(M)	0.0217	0.0040	0.0860	0.95	42 \pm 5	1-2
064C-DA	0.0022	0.0009	0.0944	0.95	32 \pm 3	1-10
064C-DA34(E)	0.0740	0.0079	0.0920	0.95	71 \pm 10	1-3
064C-DA34(M)	0.0420	0.0113	0.0870	0.95	79 \pm 7	1-3

^(a) Inert gas fusion method.

^(b) Combustion extraction method.

^(c) Inductively-coupled plasma (ICP) method.

Chemical Analysis: Table II shows that there was some decrease in the O-content upon double-anneal of all the samples, and this was accompanied by a decrease in C-content in LC-DA. These losses may have been in the form of CO and/or CO₂ during the heat treatment. However, all the samples picked up O during the creep testing at 1350 K. In general, this contamination appeared to be higher in the middle regions as compared to the ends of the crept samples. The lack of oxides in phase-extracted residue indicates that O was in solid solution in the samples. There was no significant change in C or N contents during the tests. Given the long testing periods, the extent of contamination can be considered minimal.

As-Received Samples: The as-cast, as-extruded and as-rolled microstructures in Fig. 1 were typical of the other similar samples. Fig. 1(a) shows that the coarse precipitates were distributed throughout the matrix and somewhat continuously along the grain boundaries of the cast sample. Most of these precipitates were over 10 μm along their major axes. Fig. 1(b) shows that 4:1 extrusion at 1900 K caused the precipitates to break down, and align in the extrusion direction; however, needle-like precipitates, similar to those in the as-cast sample, were also present. The cold-rolled samples (Figs. 1(c,d)) had microstructures with highly-deformed grains and precipitates aligned in the rolling direction. The precipitates were more discrete and rounded, and varied in size from less than 1 μm to over 5 μm . It is evident that marked changes in precipitate morphology and distribution took place during the operations used in sheet fabrication.

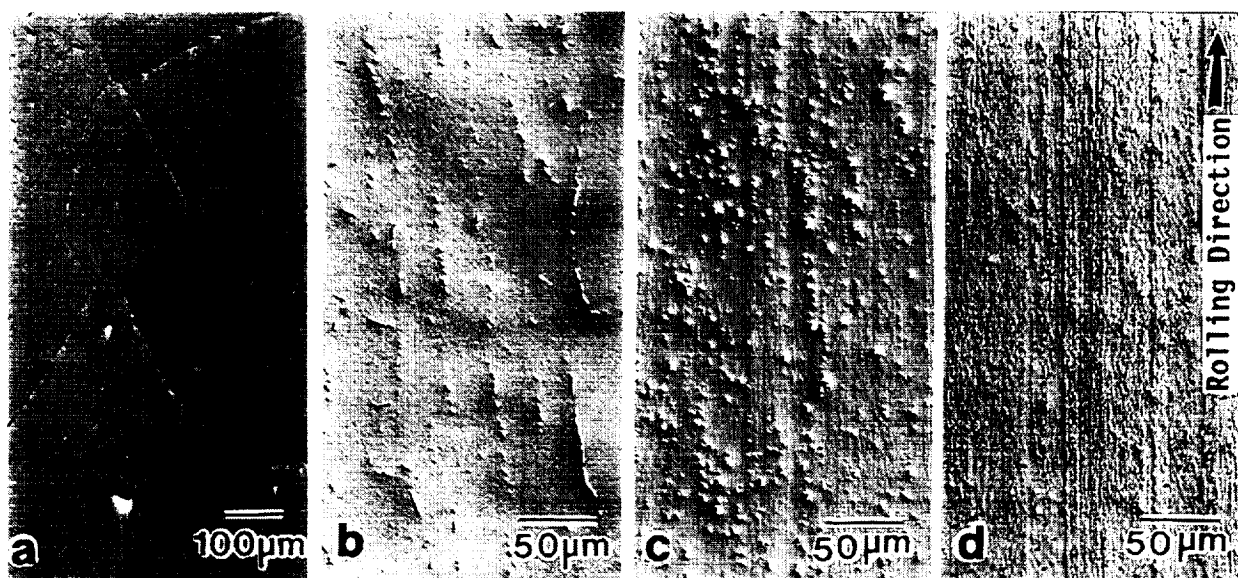


Figure 1. Microstructures of Nb-1Zr-0.1C alloy samples: (a) as-cast, secondary-electron SEM image, as-etched; (b) single-extruded bar, optical, differential interference contrast (DIC), as-polished; (c) 064A: as-rolled sheet from single-extruded bar, optical, DIC, as-polished; (d) the same as (c), but also etched.

Annealed Samples: Fig. 2(a) shows the microstructure of the double-annealed Nb-1Zr-0.06C sample (LC-DA) which appeared recrystallized. The grains were nominally equiaxed with an aspect ratio of less than 2 and an average grain size of about 25 μm (Table II). The double-annealed and aged sample (LC-DA/AGE) was very similar in appearance to Fig. 2(a) indicating ageing of LC-DA at 1350 K for 1000 h had no significant effect on its structure. However, there were noticeable differences among the microstructures of the double-annealed samples from the single-, double- and triple-extruded Nb-1Zr-0.1C sheets as are evident from Figs. 2(b-d) and Table II. Both 064A-DA and 064B-DA had relatively equiaxed grains with

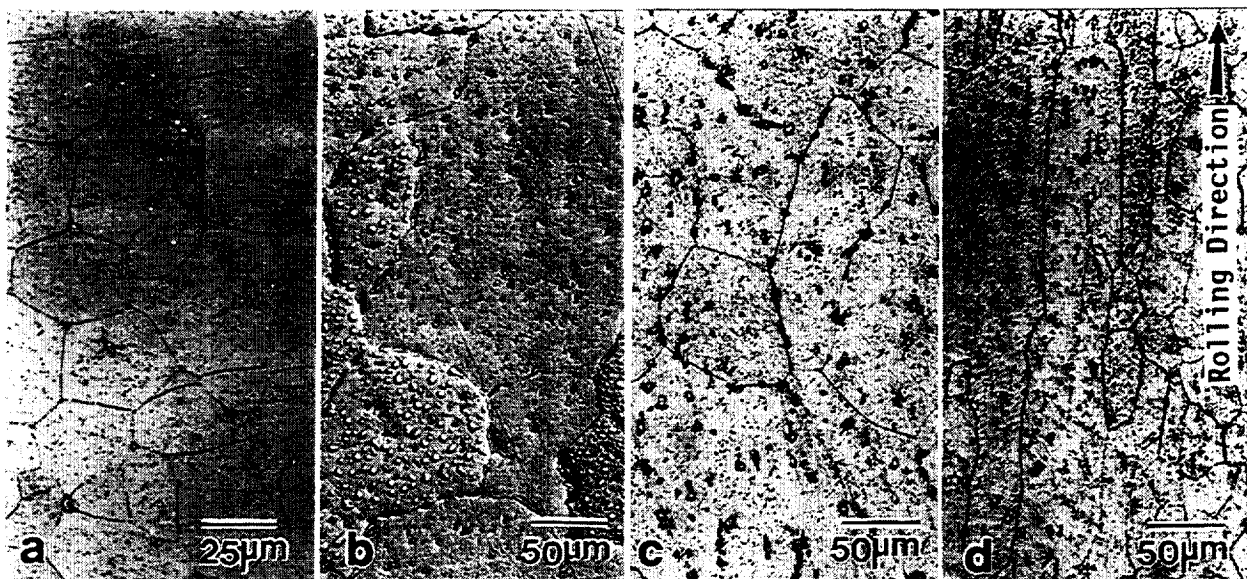


Figure 2. Double-annealed (DA: 1 h @ 1755 K and 2 h @ 1475 K) microstructures of samples from (a) Nb-1Zr-0.06C sheet LC-DA, (b) single-extruded Nb-1Zr-0.1C sheet 064A-DA, (c) double-extruded Nb-1Zr-0.1C sheet 064B-DA, and (d) triple-extruded sheet 064C-DA. All as-polished and etched, DIC.

an aspect ratio of less than 3 indicating full recrystallization, but 064C-DA had mostly elongated grains with aspect ratios varying from 1 to nearly 10. The average grain size of these samples were approximately 112, 69 and 32 μm for 064A-DA, 064B-DA and 064C-DA, respectively. These observations indicate that the degree of recrystallization and average grain size decreased with increased number of extrusions. This may be due to the marked difference in the percent cold work (%CW) in the as-rolled sheets Nb-1Zr-0.1C (Table I). The %CW in 064C (66%) was much smaller than that in either of the 064A and 064B (96 and 88%, respectively). This would lead to lower stored energy and driving force for complete recrystallization. Another possible explanation for the highly elongated grains in 064C-DA may be that the precipitates stopped the growth in the transverse direction. However, this should have been, but was not, the case in other samples. Regardless of the apparent differences in their microstructures, the precipitates, varying in size (from 1 to about 5 μm), were finely distributed throughout the matrix and along the grain boundaries in all the samples. Also, the precipitates appeared to be more abundant in the 0.1-C samples than in the 0.06-C samples as expected.

Crept Samples: Fig. 3(a-d) show the microstructures of the gage sections of samples LC-DA10, 064A-DA34, 064B-DA34 and 064C-DA34. The microstructure of the gage and low-stress end sections of each sample were similar. This was not unexpected, because the total strain in each of the crept samples was negligibly small ($< 0.3\%$). A comparison of Fig. 3(a) with Fig. 2(a) shows that the microstructure, including the grain size (Table II) of LC-DA did not change significantly during exposure at 1350 K for over 30,000 h with or without the applied stress. This was also the case for LC-DA/AGE indicating that ageing at 1350 K for 1000 h following the double-anneal did not deteriorate the excellent high-temperature stability of the microstructure of the Nb-1Zr-0.06C alloy.

In contrast to the 0.06-C sheets, changes during the creep testing of the samples from the 0.1-C sheets were more apparent as can be seen from a comparison of their respective microstructures in Figs. 2 and 3 and the grain size data in Table II. The grains of the crept samples from the single- and double-extruded



Figure 3. Microstructures of the gage sections of the samples after creep testing at 1350 K and (a) 10 MPa for 34,500 h (LC-DA10(M)), (b) 34.5 MPa for 18,780 h (064A-DA34(M)), (c) 34.5 MPa for 15,460 h (064B-DA34(M)), and (d) 34.5 MPa for 9950 h (064C-DA34(M)). All as-polished and etched, DIC.

sheets (064A-DA34 and 064B-DA34, respectively) were still relatively equiaxed, but appeared to have undergone noticeable refinement in size. The decrease in the grain size upon exposure to 1350 K and 34.5 MPa was from about 110 to 30 μm for 064A-DA after nearly 19,000 h, and from roughly 70 to 40 μm for 064B-DA after about 15,500 h. While a decrease in grain size during thermal exposure is unusual, it is possible that the low-angle boundaries were not revealed by etching in the double-annealed samples from these sheets. Subsequent long-time exposure to 1350 K, however, could have resulted in the exposure of such boundaries as a result of solute segregation and carbide precipitation. The changes in the microstructure of 064C-DA were more as expected upon creep testing for nearly 10,000 h at 1350 K. (Figs. 2(d) and 3(d)). The grains were highly-elongated with an average size of about 31 μm prior to the test, but became fairly equiaxed with over a 2-fold increase in size (Table II). This would indicate that grain growth, especially in the transverse direction, occurred in this sample during the prolonged exposure to 1350 K.

In order to verify that the protrusions and/or pits in the as-polished and/or etched samples were indeed precipitates rather than optical artifacts, the samples were also examined via an SEM. Representative results can be seen from Fig. 4 which shows the back-scattered electron images of samples from the single-extruded sheet before and after creep testing, (064A-DA and 064A-DA34(M), respectively). The precipitates, which are carbides of Nb and/or Zr, appear darker in these images, because they have a lower average atomic number than the Nb-Zr matrix. While the carbides varied in size, and were rather coarse and somewhat continuous along the grain boundaries of 064A-DA, they were a micrometer or less and finely-distributed throughout the matrix in 064A-DA34(M).

Work on Phase-Extracted Residue

The results of the analyses of the phase-extracted residues are tabulated in Table III. The precipitates in the as-rolled Nb-1Zr-0.06C (LC) sheet were orthorhombic Nb_2C , and the residue analyzed nearly all Nb. In the double-annealed sample LC-DA, some cubic phase, $(\text{Zr},\text{Nb})\text{C}$ was detected together with Nb_2C ,

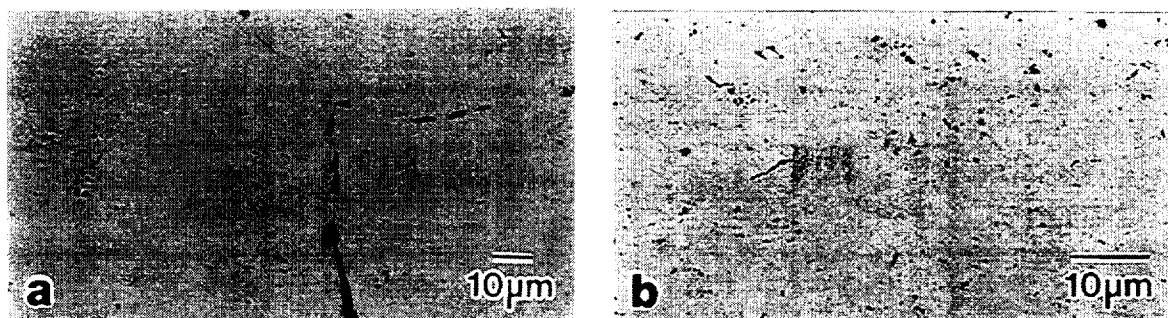


Figure 4. Back-scattered SEM images of samples from single-extruded Nb-1Zr-0.1C sheet (a) as double-annealed (064A-DA), and (b) after creep testing at 1350 K and 34.5 MPa for 18,780 h (064A-DA34(M)).

and this was accompanied with a very small increase in Zr/Nb ratio. The only phase detected in LC-DA/AGE was the cubic carbide with a_0 of about 0.460 nm indicating that ageing of the double-annealed sample at 1350 K for 1000 h resulted in conversion of all Nb_2C to $(Zr,Nb)C$. This was also accompanied by a significant increase in Zr/Nb ratio from ~ 0.04 to ~ 0.6 . The only phase detected in the crept samples from the Nb-1Zr-0.06C alloy was $(Zr,Nb)C$ with a Zr/Nb ratio of greater than 1. This indicates that exposure to 1350 K for over 30,000 h did not change the phase make-up of LC-DA/AGE, while it resulted in complete transformation of Nb_2C to $(Zr,Nb)C$ in LC-DA, possibly during the first 1000 h. The changes in the phase make up of Nb-1Zr-0.1C samples (064- in Table III) during high temperature exposure were similar to those of Nb-1Zr-0.06C. The only type of precipitate detected in the residue of the as-cast ingot was Nb_2C which again analyzed nearly all Nb indicating that Zr was in solid solution. High temperature extrusions prior to cold rolling, subsequent double-anneal heat treatment and prolonged exposure to 1350 K evidently resulted in increasing transformation of Nb_2C to $(Zr,Nb)C$. This is evident from the increased detection of the cubic phase and the accompanying increase in the Zr/Nb ratio along the process path of each of the samples in Table III. Again, the only phase detected in the residue from the crept samples was the cubic $(Zr,Nb)C$ indicating complete transformation of Nb_2C to the more stable $(Zr,Nb)C$. Another noteworthy trend observed was that the Zr/Nb ratio, which is an indicator of the relative abundance of the cubic carbides, increased with increasing number of extrusions (Table III). This trend was reinforced by also analyzing the residue from the double-annealed Nb-1Zr-0.1C sheets by energy-dispersive x-ray spectroscopy using SEM as can be seen from the last column in Table III. Furthermore, the identification of the precipitates by x-ray and chemical analysis of the residue has been verified by TEM studies²³. In 064A, the precipitates were determined to be orthorhombic Nb_2C regardless of size which varied from 1 to about 5 μm . In the double annealed samples, the coarser precipitates were again Nb_2C , but the finer precipitates (1 μm or less) were cubic $(Zr,Nb)C$. These cubic carbides were coherent with the matrix and were finely-distributed in the samples exposed to 1350 K for very long times.

The lattice parameters, a_0 , of the cubic phase in Table III, 0.450-0.468 nm fall between the a_0 values of NbC (≈ 0.447 nm) and ZrC (≈ 0.470 nm). As both carbides have FCC crystal structures, it is probable that the cubic precipitates are solid solutions of NbC and ZrC. As the phase transformation from Nb_2C to $(Zr,Nb)C$ increased, the Zr/Nb ratio in the residue of a sample also increased and was generally accompanied by an increase in the a_0 value of the cubic carbide. This would be expected, because $a_0(Zr) > a_0(Nb)$ and $a_0(ZrC) > a_0(NbC)$. Similar a_0 values and Zr/Nb ratios for $(Zr,Nb)C$ as those in Table III have also been reported in the literature^{11,18-23}. It has also been suggested that the solid

Table III. Results of the analysis of phase-extracted residue from Nb-1Zr-0.06C (LC-) and Nb-1Zr-0.1C (064-) samples.

SAMPLE	X-RAY ANALYSIS				Zr/Nb RATIO	
	PHASES	LATTICE PARAMETERS (nm)			CHEM ANALYSIS	SEM ^(a) XEDS
		a ₀	b ₀	c ₀		
LC	Nb ₂ C	1.09	0.492	0.311	1/99	
	(Zr,Nb)C	0.468				
LC-DA	Nb ₂ C				4/96	
	(Zr,Nb)C	0.458				
LC-DA10(E)	(Zr,Nb)C	0.465			72/27	
LC-DA10(M)	(Zr,Nb)C	0.464			63/37	
LC-DA/AGE10	(Zr,Nb)C	0.460			38/62	
LC-DA/AGE10(E)	(Zr,Nb)C	0.465			60/40	
LC-DA/AGE10(M)	(Zr,Nb)C	0.465			56/44	
064-INGOT	Nb ₂ C	1.092	0.498	0.310	2/98	11/89
064A	Nb ₂ C	1.092	0.497	0.311	3/97	
	(Zr,Nb)C	0.453				
064A-DA	Nb ₂ C	1.09	0.48	0.29	10/90	
	(Zr,Nb)C	0.450				
064A-DA34(E)	(Zr,Nb)C	0.454			58/42	
064A-DA34(M)	(Zr,Nb)C	0.454			54/46	
064B	Nb ₂ C	1.09	0.499	0.311	14/96	
	(Zr,Nb)C					30/70
064B-DA	Nb ₂ C				37/63	
	(Zr,Nb)C	0.459				
064B-DA34(E)	(Zr,Nb)C	0.456			63/37	
064B-DA34(M)	(Zr,Nb)C	0.454			57/44	
064C	Nb ₂ C	1.09	0.497	0.311	25/75	
	(Zr,Nb)C					
064C-DA	(Zr,Nb)C	0.459			63/37	
064C-DA34(E)	(Zr,Nb)C	0.459			72/28	71/29
064C-DA34(E)	(Zr,Nb)C	0.458			85/15	

(a) Average of analysis of 4 areas ranging in size 250x250 μm to 5x5 μm .

solutions of NbC and ZrC yield non-stoichiometric carbides of $(\text{Zr,Nb})\text{C}_x$ or $\text{Zr}_x\text{Nb}_y\text{C}_z$ with x and y varying between 0.8 and 0.98, but this could not be verified in this study. However, it is evident from the results of this study that the finely distributed cubic carbides of $(\text{Zr,Nb})\text{C}$ form as a result of transformation from Nb_2C during thermomechanical processing and/or exposure to elevated temperatures. This transformation has been reported to be complete from about 100 h¹⁹ to over 10,000 h¹⁸ of ageing at temperatures over 1300 K. The results in this study show that the transformation of Nb_2C to the stable monocarbide was complete within the first 1000 h of exposure to 1350 K in the Nb-1Zr-0.06C alloy. In the Nb-1Zr-0.1C sheets, it appeared to be completed during the double-anneal in the triple-extruded sheet, 064C-DA, and may have been completed in the others early during the exposure to 1350 K with or without applied stress.

CONCLUSIONS

The results presented in this paper clearly show that the rather coarse Nb_2C , which is orthorhombic and forms during casting of Nb-1Zr-C alloys, increasingly transforms to submicron-sized cubic (Zr,Nb)C during thermomechanical processing and/or high temperature exposure. The transformation of Nb_2C to (Zr,Nb)C was essentially complete in a Nb-1Zr-0.06C sheet within 1000 h at 1350 K following a double-anneal heat treatment (1 h @ 1755 K + 2 h @ 1475 K). The phase transformation was complete also during the double-anneal in the triple-extruded sheet and during the subsequent 1350-K exposure in the single- and double-extruded sheets from the Nb-1Zr-0.1C alloy. Once formed, the cubic carbides were extremely stable giving the alloy excellent microstructural stability during prolonged (10,000 - 34,500 h) exposure to 1350 K with or without applied stress.

Increasing the C-content from 0.06 to 0.1 wt.% resulted in an increase of the amount of precipitates as be expected. The multiple extrusion of Nb-1Zr-0.1C alloy at 1900 K affected the double-annealed microstructures, but appeared to have no end-benefit concerning high temperature stability. The microstructures of the samples from the single-, double - and triple-extruded sheets all appeared similar with the cubic phase finely-distributed throughout the matrix.

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REFERENCES

1. R.H. Cooper, Jr., in Refractory Alloy Technology for Space Nuclear Power Applications, edited by R. H. Cooper and E. E. Hoffman, (Technical Information Center, US Department of Energy, Oak Ridge, TN 1984) pp. 14-17.
2. R. H. Titran, Advanced Materials and Processes **142**(5), 34-41(1992).
3. G. C. Kruger, S. Vaidyanathan, N. Deane, R. Protsik, R.E. Murata, in SP-100 Reactor Design, (22nd Intersociety Energy Conversion Engineering Conference Proc. 1, AIAA, New York, 1989) pp. 419-423.
4. E.J. DelGrosso, C.E. Carlson and J.J. Kaminsky, JLCM **12**, 173-201(1967).
5. W. Dokko, JPL Report No. JPL D-1948, Jet Propulsion Lab., Pasadena, CA, 1984.
6. A. J. Bryhan and R. C. Chan, JOM **45**(6), 50-53(1993).
7. R.H. Titran, NASA TM-100142, NASA LeRC, Cleveland, OH, 1986.
8. R.H. Titran, T.J. Moore and T.L. Grobstein, NASA TM-89834, NASA LeRC, Cleveland, OH, 1987.
9. R.H. Titran, NASA TM-102390, NASA LeRC, Cleveland, OH, 1990.
10. Mehmet Uz and R.H. Titran, NASA TM-106319, NASA LeRC, Cleveland, OH, 1993.
11. J.T. Norton and A.L. Mowry, J. Metals **1**(2), 133-136(1949).

12. F. B. Cuff, Jr., Report ASD-TDR-62-7, PART I, Wright Patterson AFB, OH, 1962.
13. R.T. Begley, R.L. Ammon, and R. Sticker, Report WADC-TR-57-344, PART VI, Wright Patterson AFB, OH, 1963.
14. F. Ostermann and F. Bollenrath, AFML-TR-66-259, Wright Patterson AFB, 1966.
15. F. Ostermann, J. Less-Common Metals, 25, 243-256(1971).
16. A.E. Kissil, P.A. Khandarov, A.D. Rapp, L.P. Onisenko, L.Z. Polyak, A.G. Arakelov and I.I. Maximow, Sov. Mater. Sci., 12, 640-643(1976), (Engl. Transl.)
17. V.B. Arsomosov, and E.V. Vasil'eva, Met. Sci. Heat Treat., 20, 291-293(1978),
18. P.A. Khandarov, A.N. Luk'yanov, A.G. Arakelov, O.S. Tsvikilevich, E.M. Lyutyi and G.G. Maximovich, Sov. Mater. Sci., 14, 431-435(1978), (Engl. Transl.)
19. A.D. Korotayev, A.N. Tyumentsev, M.G. Glazunov, L.M. Dizhar, A.I. Yakushina, S.P. Semkin and T.I. Vitkovskaya, Phys. Met. Metallogr., 52, 128-135(1981).
20. G.K. Moiseev, N.A. Vatolin and S.K. Popov, Izv. Akad. Nauk SSSR, Neorg. Mater., 19(9), 1478-1483(1983). (Engl. Transl.)
21. T.L. Grobstein and R.H. Titran, NASA TM-100848, NASA LeRC, Cleveland, OH, 1986.
22. Mehmet Uz and R.H. Titran, NASA TM-103647, NASA LeRC, Cleveland, OH, 1991.
23. Mehmet Uz and R.H. Titran, 10th Symp. Nuclear Power and Propulsion, AIP Conf. Proc. 271, edited by M.S. El-Genk and M.D. Hoover, Albuquerque, NM 1993, pp. 69-83.
24. ASTM Standard E112, Annual Book of ASTM Standards 03.03, ASTM, Philadelphia, 120(1984).

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13. ABSTRACT (Maximum 200 words) High temperature stability of the microstructure of Nb-1Zr sheet containing 0.1 and 0.06 wt.%C was studied as affected by processing and prolonged 1350-K exposure with and without applied stress. Sheets were fabricated by cold rolling bars that were single-, double- or triple-extruded at 1900 K. Creep samples were double-annealed (1 h @ 1755 K + 2 h @ 1475 K) prior to testing at 1350 K and 10,000 - 34,500 h. The microstructures of the as-cast, extruded, rolled, DA and crept samples were characterized using various metallographic and analytical methods. The precipitates were rather coarse Nb ₂ C initially, but transformed to finer ($\leq 1 \mu\text{m}$) carbides of (Zr, Nb)C with each subsequent high temperature process. The grain size, and the relative amount and morphology of (Zr, Nb)C were found to be affected by the number of extrusions and to some extent by C-content. However, the microstructures of all the crept samples were similar with (Zr, Nb)C distributed throughout the matrix indicating that prolonged exposure to 1350 K gave rise to complete transformation of Nb ₂ C to (Zr, Nb)C regardless of the processing history. These and other observations are presented with the emphasis on the correlation between processing, microstructure and creep properties.				
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